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Evaluation of Non-Destructive Techniques for Monitoring Material Degradation

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List of abbreviations and symbols

Partners

- EDF: Électricité de France
- NRG: Netherlands Energy Research Foundation
- VTT: Technical Research Centre of Finland
- Tecnatom: Tecnatom SA
- IZFP: Fraunhofer-Gesellschaft zur Förderung der Angewandten Forschung e.V.
- JRC: Joint Research Centre
- Serco: Serco Assurance
- CIEMAT: Centro de Investigaciones Energeticas, Medioambientales y Technologicas
- ARCS: Österreichisches Forschungscentrum Seiberdorf Ges.m.b.H.
- AEKI: KFKI Atomic Energy Research Institute
- Uni. Han.: University of Hannover
- PSI: Paul Scherrer Institut
- NRI: Nuclear Research Institute REZ
- SPG: Siempelkamp Prüf- und Gutachter Gesellschaft MbH
- FANP: Framatome ANP

Subcontractor

- INSA de Lyon: Institut National des Sciences Appliquées de Lyon (subcontractor to EDF)

- α': martensite phase
- LCF: Low Cycle Fatigue
- Nf: number of cycle that marks the end of the fatigue testing
- noc: number of cycle (for fatigue testing)
- D: usage factor Ni/ Nf
- R: ratio between tensile and compressive strain

Non-destructive techniques

- RRT: Round Robin Test
- NDT: Non Destructive Testing
- TEP: ThermoPower Measurements
- STEAM: Seebeck and Thomson Effect on aged material
- MAG: Micromagnetic measurement
- MBN: Magnetic Barkhausen noise or BNA: Barkhausen Noise Analysis
- BN: Barkhausen Noise
- NLH: Non-Linear Harmonic analysis of magnetic signals
- ABIT: Automated Ball Indenter
- GMR: Giant Magnetic Resistor
- USS: Ultrasonic Scattering
- ULRW: Ultrasonic Leaky Raleigh Wave technique
- 3-axis fluxgate: Remanence field measuring with fluxgate
- Ferromaster: Permeability measuring with Ferromaster
- HAS: Harmonic Analysis of magnetic signals System
- ND: Neutron Diffraction

Executive publishable summary

The lifetime management of ageing power plants for electricity production is an economical way to reduce the electricity generating costs for the benefit of the customers. Managing the lifetime of existing installations requires the development of innovative reliable techniques for the inspection of critical components. Such techniques will detect changes in the materials and will allow planning actions for failure prevention, e.g. changing operation parameters, increase inspection intervals or replace components.

The main objective of the GRETE project is to assess the capability and the reliability of innovative inspection techniques by means of a round-robin exercise. The non-destructive techniques that are tested are different from standard inspection methods. The aim of standard techniques is to detect macroscopic defects like cracks, including for certain applications sizing and imaging. The methods applied in this project are sensitive to any microstructural change in the material leading to a degradation of the mechanical properties of the component long before macroscopic cracks are initiated and eventually grow.

For the GRETE project, two aging mechanisms were chosen to illustrate the potential use of NDT techniques for the monitoring of material degradation. One is the neutron irradiation damage occurring in reactor pressure vessels made of ferritic steels and the other is the fatigue affecting austenitic stainless steel piping:

- The monitoring of irradiation damage by non-destructive techniques would limit the destruction of the specimens of surveillance programme and make possible their re-insertion in the reactor for further regular non-destructive measurements. Samples from irradiation surveillance programme or experimental reactor of four countries have been chosen and collected in the hot cells facility of NRG (Petten, the Netherlands).

- The monitoring of fatigue damage by non-destructive techniques would prevent any unexpected leakage by the early detection of any significant microstructural change. Specimens from three different austenite steel grades with adapted geometry have been tested in low cycle fatigue. The parameters of the low cycle fatigue tests have been chosen to create a whole range of fatigue damages. The microstructural changes related to fatigue damage (dislocation network and martensitic phase) have been observed using Transmission Electron Microscopy, Neutron Diffraction and advanced X-Ray Diffraction methods.

For the two damage mechanisms round robins have been organised. The evaluation of the non-destructive techniques has been performed in each laboratory participating to the testing by comparing precisely the non-destructive signal measured and the material characteristics. The results of the testing have been gathered and interpreted in terms of sensitivity to the detection of damages. The different techniques have been compared on their different capabilities.

The results have been used for drawing recommendations for the use of non-destructive techniques in monitoring on site components degradation.

A. OBJECTIVES AND SCOPE

Materials are ageing due to industrial conditions: high pressure and temperature, corrosion atmosphere, irradiation, etc. Microstructures of materials evolve: vacancies generation and dislocations, precipitation on the grain boundary, etc. This evolution leads to an embrittlement of the materials by decreasing their resistance to cracking. The assessment of material ageing is normally done by destructive testing and/or investigations of samples. These operations are very expensive and in some cases cannot be applied on a specific component. That is why the development of non-destructive evaluation techniques to evaluate material degradation may be an interesting alternative. These non-destructive evaluation techniques are different from standard inspection methods. The methods applied in this project are sensitive to any microstructural change in the material leading to a degradation of the mechanical properties of the component long before macroscopic cracks are initiated and eventually grow.

This way of thinking has been shared by many utilities and research centres, and a lot of studies have been launched. Nevertheless, up to now, few real industrialised non-destructive evaluation methods have been applied on site. There exist difficulties to interpret the non-destructive signals measured in term of ageing parameter and most of the non-destructive evaluation techniques are still under development and not yet qualified.

In this context, a 16 months concerted action, AMES-NDT, has been launched within the Fourth Framework Programme in 1998. Its objectives were to compile all known NDT techniques used in the EU, in the US, in Japan and in Russia and to carry out demonstration tests for some of these techniques. These tests have been successful and the results promising, but most of the techniques studied were still under development. Further research was needed in order to validate and qualify NDT techniques and to apply them to monitor ageing in real components.

In this context, the shared cost action GRETE was launched in the Fifth Framework Programme. The main objective of the GRETE project is to assess the capability and the reliability of innovative inspection techniques by means of a round-robin exercise. The partners using various techniques test aged samples. In order to correlate the changes in the measured NDT signals to the microstructural changes in the material due to ageing, careful microstructural examinations of the material damage are done.

For the GRETE project, two ageing mechanisms were chosen to illustrate the potential use of NDT techniques:

- The monitoring of irradiation damage by non-destructive techniques would limit the destruction of the specimens of surveillance programme and make possible their re-insertion in the reactor for further regular non-destructive measurements.
- The monitoring of fatigue damage by non-destructive techniques would prevent any unexpected leakage by the early detection of any significant microstructural change.

The GRETE project aims at developing quantitative information on the ability of innovative non-destructive to monitor aging. Moreover, the GRETE project plans to pave the way of new non-destructive techniques applied to most of the important degradation phenomena in critical components.

B. WORK PROGRAMME

The project work-plan is composed of eight work packages. Three of them (WP1, WP4 and WP5) are related to the work on neutron irradiation damage, three (WP2, WP3 and WP6) are related to the work on fatigue damage and the last two (WP7 and WP8) are common to both works. Finally, with reference to the objectives of this project, the structure of the work plan may be broken down in (1) the preparation of the testing [WP1, WP2, WP4], (2) the evaluation of NDT techniques [WP5 and WP6] and (3) the understanding of NDT results [WP3, WP7 and WP8]. The synthetic organisation of the work packages is given in Figure 1.

B.1 WP 1: Providing and preparation of the irradiated samples

WP1 concerns the providing and the preparation of the irradiated samples that have been measured by non-destructive techniques. Four partners have proposed irradiated specimens of RPV steel.

B.2 WP 2: Providing and preparation the samples for the fatigue tests

Three grades of austenitic stainless steel, widely used in power generating systems for piping and support structures, have been provided. During this work package the partners have chosen: the material of the fatigue specimens, the geometry of the specimens and the parameters of the fatigue testing. The specimens have been have been Low Cycle Fatigue (LCF) tested by EDF (INSA de Lyon), PSI, SPG and FRANP.

B.3 WP 3: Characterisation of the LCF samples

One of the major difficulties in the analysis of the non-destructive measurements is to clearly quantify the influence of each microstructure parameters on the non-destructive signals measured. So it is important to have destructive investigations on the measured specimens in order to clearly understand the evolution of the microstructure in relation to the measured signal. The partners (PSI and INSA de Lyon) have observed the microstructural changes (dislocation network and martensitic phase) related to the fatigue damage.

B.4 WP 4: Preparation of the testing on irradiated specimens

Measurements on irradiated specimens may be difficult. In this context, each partner has been responsible for the adaptation of its measurement device to hot cells

B.5 WP 5: Non destructive testing on irradiated materials

This work package corresponds to the organisation of the round-robin tests on the irradiated specimens. All the measurements have been performed in NRG's hot cells. NRG team has supervised every partner in order to perform the measurement in good conditions.

B.6 WP 6: Non destructive testing on LCF specimens

This work package corresponds to the organisation of the round-robin test on the Low Cycle Fatigue specimens.

B.7 WP 7: Analysis and interpretation the NDT results (WP5 and WP6)

The data obtained from the testing has been compared to the results of the microstructural investigations. The evolution of the microstructural changes has been related to that of NDT signals. As a consequence, the sensitivity of each technique to detect and to quantify the material damages has been evaluated. Quantitative criteria (accuracy, reproducibility, dispersion of data) have been set and used to characterise the performance of each technique.

B.8 WP 8 Recommendations

This work package aims at drawing recommendations on the use of non-destructive techniques in an industrial context. Recommendations are based on the results of the non-destructive measurements of The GRETE project but also on the state of the art for each technique.

C. WORK PERFORMED AND RESULTS

C.1 Building of reference specimens sets

C.1.1 Preparation of the irradiated specimens set (WP1)

C.1.1.1 Supplying the irradiated specimens

The irradiated specimens have been taken out of surveillance programmes of irradiation and research programmes. The objective was to have a large range of fluence representative of the as received specimens up to the irradiated specimen with a received fluence close to 40 years of reactor activity. The characterisation of the specimens (fracture toughness transition behaviour, tensile properties, Charpy impact transition …) has been done before the GRETE project. The irradiated samples are broken Charpy V notch test specimens. All the samples are chosen in the low temperature range of the transition curve (brittle domain) to avoid local strains which could be present in the samples.

C.1.1.2 Preparation of the specimens for the measurements

The surfaces were ground with a 1000 grid and then cleaned with alcohol. During the round-robin test of non-destructive measurements of irradiated specimens, the quality of the surface preparation of the specimen has been checked and maintained because the manipulation of the specimens in the hot cell may damage the surface polishing. The face which was polished was the one with the notch in order to limit the plastic deformation effect on the measurements.

C.1.2 Preparation of the Low cycle fatigue specimens (WP2)

C.1.2.1 Characteristics of the materials tested

Three different austenitic materials were chosen for the LCF-tests: AISI 304L, AISI 321 and AISI 347 (corresponding to 1.4401; 1.4541 and 1.4550 of German Standard DIN EN 10088-3 (former DIN 17440)). The non-stabilised AISI 304L material was tested in the “as received” and in the “cold-worked” condition (laminated, 33 % reduction of thickness) in order to consider the influences of processing. For the Ti-stabilised AISI 321 and the Nb-stabilised AISI 347 materials, both manufactured according to DIN 17440, only the “as received” condition was investigated. The austenitic stainless steel gets a solution heat treatment (1000 to 1120 °C, quenching in water or fast in air) according to DIN EN 10088- 3 as the final heat treatment.

C.1.2.2 The samples

The final surface quality with a depth of roughness of less than 0.2 µm is necessary to avoid an early crack initiation. The non-destructive investigations are concentrated on the 20 millimetres long polished cylindrical gauge length of the specimen (see **Figure 2**).

C.1.2.3 Test Parameters

The fatigue tests were achieved under total strain control. It has been decided to choose a total strain range of ± 0.4 % for almost all fatigue tests. This is a compromise taking into account the operational loading of components which is normally in the range of lower yield strength and the fact that higher strain amplitudes cause a higher content of strain induced martensite. The Usage Factor (D) is defined as the ratio between the actual number of load cycles N_i supplied by a specific specimen i and the average number of cycles N_f for the fixed end of life, which was defined as a drop of 2% in the maximum stabilised force curve after having passed the hardening/softening phases.

C.1.3 Destructive investigation of the LCF specimens (WP 3)

C.1.3.1 Experimental matrix for the characterisation of the fatigue specimens

The examination methods for the characterisation of the specimens are listed below:

- Inductive Coupled Plasma Emission Photometry (ICP-OES) for the determination of the chemical composition.
- Neutron- and advanced X-ray diffraction methods for the quantitative determination of martensite.
- Scanning electron microscopy (SEM) for the observation of cracks, slip bands between grain and twin boundaries.
- Transmission electron microscopy (TEM) for the observation of crystal defects.
- Metallographic investigations for the determination of shape and size of grains and martensite.

Based on results from former investigations, main attention has been paid to the content of martensitic phase as an indicator for microstructure evolution.

C.1.3.2 Determination of martensitic content

Since most NDT-methods are considered as indirect methods for the detection of martensite, neutron diffraction was applied as a reference method for a quantitative determination of martensite. For all materials a linear dependency between the number of cycles (noc) and martensitic content was found if strain induced martensitic transformation occurs at all. Large differences in martensitic contents in the final state between the investigated materials were observed, even if the noc which led to crack initiation did not deviate significantly. At the temperature of 300°C only a small amount of α' was generated, this is in agreement with other experiments and theoretical aspects.

C.2 Round-robin tests (WP5, WP6)

C.2.1 Round-robin tests on irradiated specimens (WP5)

The principle of the round-robin test was a blind test with reference specimens. It has been decided by the partners to make measurements without knowing the properties of the samples. Nevertheless, it was impossible to have a completely blind round-robin test for two main reasons: it was impossible to remove the original marks of the samples and change it by a neutral mark, and some non destructive testing need to be calibrated with reference specimens. Five different techniques have been applied on the irradiated specimens. They are listed in Table I.

C.2.2 Round-robin test on Low Cycle Fatigue specimens (WP6)

Special boxes have been manufactured in order to facilitate the storage of the samples and their transportation from partners to partners. The round robin has been a blind one except for one set of EDF specimens, because some techniques need calibration specimens. Eight different techniques have been used on the low cycle fatigue specimens. They are listed in Table II.

C.3 Analysis of the Non-Destructive Measurements (WP 7)

C.3.1 Non destructive measurements on irradiated specimens

C.3.1.1 STEAM (JRC)

STEAM is a non-destructive method specially developed at the JRC-IE to assess the embrittlement state of materials. STEAM is based on the measurement of the Seebeck coefficient, characteristic of the material and related to its microstructural arrangement. In general results were stable and repeatable. The STEAM non-destructive technique is a method for qualitative analysis: the judgement about the state of ageing of a certain material under test depends on the comparison between the value for the “aged” condition and the baseline value.

In summary, good correlation between STEAM and material properties have been found for most of the groups of samples and irradiation conditions. A correlation exists between the accumulated fluence and the shift in the STEAM value. A linear correlation between shifts in STEAM and Transition Temperature has been found. STEAM can be compared with hardness measurements, the same trend has been found for both of them: irradiation increases the hardness as well as the STEAM value.

C.3.1.2 3MA (IZFP)

IZFP has applied micro-magnetic techniques to characterise the neutron embrittlement. These techniques in general are sensitive for irradiation damage because the radiation – induced lattice defects are vacancies and fine-dispersed Cu-precipitates in the 1-2 nm diameter range. There is a strong interaction between these precipitates and the magnetic Bloch-walls of the domains by pinning effects and by residual stress effects. 3MA is based on multiple parameter regression algorithms modelling target functions which are in this case the fluence or the transition temperature. By screening a full set of specimen optimal 3MA test parameters were evaluated.

In order to test the multiple regression result some of the specimens were selected as test specimens and the regression was performed again using the residual set of calibration specimen. The reproducibility is high. However, some test samples are documented out of the regression model range because it is indicated with negative fluence values. This shows, that in this specimen a strong influence parameter is present – may be the residual stress effect mentioned above – which is not considered in the calibration.

In the case of the fluence as target-function the regression algorithm stops with a regression coefficient of R = 0.922 and a standard error of estimate of 1.188 fluence units (10^{19} n/cm^2). However, also the test with independent selected test specimens reveals under

- as well as over - estimation of the material microstructure states. It is obvious that the scatter in the data seems to be larger for low and medium-low fluence values

It was the first time that a 3MA-approach was applied at irradiated specimen. It was becoming clear that the magnetic properties selected with 3MA are very strongly influenced by the residual stresses induced by the plastic deformation in the broken Charpy test specimen. It was found that this influence is much stronger at the specimen side with the V-notch because of the counter bearings of the Charpy hammer than of the side which is hit by the hammer. However, even on this side residual stress effects are detected.

Principally sufficiently good correlations with regression coefficients which are high enough and with small standard errors of estimate can be achieved even if the independent test with test data is not fully convincing because of the residual stress influences.

C.3.1.3 MBN (CIEMAT)

CIEMAT has applied Barkhausen noise measurements. The possibilities of several analyses have been evaluated to detect the effect produced by neutron irradiation in a set of structural steels. Based on the correlation degree found with the mechanical damage indicators in these steels, several analyses techniques have been used: avalanches, classification of pulses, hysteresis loop, PSD (power spectrum density), RMS (root means square) &H, RMS wavelength, JTFA (Joint Time Frequency Analysis). To sum up the more promising results: for Hysteresis loop, the observed tendency is that the neutron radiation in general simultaneously reduces the steel magnetic susceptibility and increases its coercitive field value. For PSD (Power spectrum density), it has revealed the existence of characteristic frequency peaks that appear or disappear by irradiation. The obtained results are very interesting and suggest new expectations for specific microstructural defects detection. However, there are many uncertainties to clarify, as well as the possibility of combining this technique with magnetoacoustic and/or ultrasonic emission. For RMS (Root means square) &H, in general, the observed tendency is the decreasing in peak height value based on the received irradiation dose. In this analysis, it is interesting to emphasize the capacities to detect different phases or microstructural aggregates. The observed correlation is good.

To conclude, it is necessary to emphasize that although the analyses of the measurements during the GRETE project suffer from sufficient number of statistical data to assure a comparable accuracy and repeatability, and of which they can be deeply affected by plastic deformations associated to the indentations previously undergone by the specimens, the obtained results throw a reasonable hope as far as the capacities of the technique and their possibilities for microstructural defects detection.

C.3.1.4 MBN (AEKI)

AEKI has performed Barkhausen noise measurements. The broken half Charpy specimens could be easily manipulated and tested in hot cell. The probe developed for this purpose is simple and easy to use it. The coupling was generally good, and it allows in the future extending the testing with magnetic permeability measurement. The permeability is more sensitive for the irradiation effect than the Barkhausen noise, and the combination of the two measurements may provide better results. The specimens giving different results on the notch and backsides have been evaluated very carefully. The different results on the two sides indicate the presence of oxide layer or residual stresses. Most probably the oxide layer arose during irradiation, and the residual stresses are the results of the impact testing. The scan of the full excitation range and the comparison of the RMS Barkhausen noise versus excitation functions seem to be good indicator of the irradiation. Maybe the comparison of the slope of the curve will provide more sensitive solution, but the evaluation methodology has to be developed.

Half of the evaluation were correct that means that the qualitative value of fluence have been found, in case of 16 specimens the evaluation error was one irradiation level, and in the case of 6 specimens the evaluation error were large. The reason of the errors was mainly the deformation of the specimens and the surface layers.

C.3.1.5 ABIT (NRI)

NRI has performed ABIT measurements on half of the specimens. The plastified volume under the indenter is surrounded by elastically deformed zone; therefore the whole indentation diagram depends on stress determined by elasto-plastic behaviour of the tested material in the range including all small nonzero values of plastic strain. Thus plastic behaviour near initiation of plastic flow, i.e. initial strain hardening rate or extent of Lüders strain, influences largely both yield stress and ultimate tensile strength obtained by "universal" calibration which does not take these nuances into account. Some systematic differences between tensile properties and ABIT results can be interpreted as consequences of both chemical composition and microstructural state of steel. Both chemical composition and microstructural state determine concentration of carbon in solid solution in ferrite and consequently extent of Lüders strain and corresponding constraint factor; if the constraint factor in calibration relations is fixed (or, equivalently, if calibration constants are fixed), systematic differences between tensile properties and ABIT results may result.

Accuracy of preliminary analysis of results obtained by examination of indentation testing method within the framework of GRETE project is limited by missing information concerning details of stress-strain behaviour of tested materials.

C.3.1.6 TEP (EDF)

The results of TEP measurements on the irradiated specimens have been compared with the fluence values and transition temperature. The main observation is that monotonous relationships have been established between TEP and fluence, transition temperature, the FIS formula (prediction of ΔRTndt), and hardness. Relationships of the same kind should be found between TEP and yield strength (a monotonous correlation may be found between yield strength and transition temperature). A relationship common to all the sets has not been determined for TEP and fluence: it seems that every set has a different behaviour. On the contrary, it seems that a relationship common to most of the sets may be found for TEP and transition temperature.

TEP is sensitive to any changes in the microstructure that have an influence on the transportation of valency electrons. That is why the correlation between TEP and transition temperature is better that the correlation between TEP and fluence. Indeed fluence cannot be directly measured on the material properties. Only the consequences of the neutrons embrittlement can be measured on the material.

C.3.1.7 HAS (Uni. Han.)

The Harmonic-Analysis-System HAS is equipped with adapted sensors, that enabled to perform the tests very easy and within a second. Several HAS are in industrial use for material characterisation.

The results of the investigations on the neutron embrittled half Charpy specimens with the Harmonic-Analysis-System show, that the state of not irradiated specimens can be clearly separated from irradiated specimens. But the measured values show high divergences for specimens with the same state of irradiation. The reasons for this are differences in microstructure and residual stress, mainly caused by plastic deformation during the Charpy impact tests. The effect of the embrittlement of the irradiated specimen on the harmonics is probably small comparing to the effect of the influence quantities. To purify the suitability of the Harmonic-Analysis-System it is necessary to reduce these influence quantities. Therefore further investigations have to be done.

C.3.2 Non destructive measurements on LCF specimens

C.3.2.1 GMR 3MA (IZFP)

Together with WK Kaiserslautern, IZFP has developed an online monitoring technique by using GMR (Giant Magnetic Resistor)-sensors, which can be used as a magnetometer or to measure eddy current transfer impedance. Because of the phenomenon that martensite is developed localized, IZFP has determined average GMR-transfer-impedance values obtained along the middle girth path in the measuring length of the hourglass specimens. Beside the GMR-transfer-impedance measurements also 3MA tests were performed.

It was found that the automatically measured magnetism by the GMR sensor gave the clearest martensite indication. Based on these PSI results a correlation with the own averaged GMR data was performed. The own measured absolute value of the transfer-impedance is compared with the PSI results. A linear correlation can be documented showing the potential of the GMR transfer-impedance measurements. It is very clear that depending on the chemical composition of the steel grade and its segregations, the loading history and the temperature conditions, the content of the developed martensite will be different. Absolutely measured, there is no correlation between martensite content and usage factor. However, in each set of individually fatigued samples there is a strong correlation between usage factor and developed transfer-impedance.

By combining 3MA quantities in a regression-approach a strong correlation to the PSI results can be found. Independently selected test samples are used to test the approach.

C.3.2.2 MBN (EDF (INSA de Lyon))

The Barkhausen Noise measurements responses have been analysed in terms of peak height, peak position and global BN activity (RMS: Root Means Square). The fatigue induces a significant BN activity only in the AISI 321 steel. Thus, a wide hump centred on H values can be observed after fatigue for every experimental condition. In general, cold rolling seems to induce a much stronger activity than low cycle fatigue and the comparison between the quantities A_{RMS} and V'_M does not reveal a marked difference between their relative variations.

This means that the shape of the broad maximum that is characteristic of the $V_{RMS} = f(H)$ response does not change very much neither due to fatigue nor due to material.

In order to complete the results obtained in "static" conditions, additional in situ measurements have been undertaken on non cold-worked and cold-worked AISI 304L specimens. The intensity of the BN activity appears to be strongly influenced by the position of the measuring point, being enhanced by a tension stress and reduced by a compression stress. It should be also noticed that a marked difference also appears between the two possible points of measurement at $\sigma = 0$. The applied stress is not the only parameter that governs the BN activity, i.e. the plastic strain history also plays a significant role.

No simple relation between BN activity and magnetic phase contents evaluated by neutron diffraction has been found. The origin of this specific sensitivity of the BN is linked to the role of internal stresses. The role of applied stresses on BN activity of magnetic phases in the AISI 304L steel has been evidenced by in situ measurements and show an enhancement of the BN activity in tension and conversely in compression. These remarks enable to explain the surprising scatter of A_{RMS} data observed.

C.3.2.3 MBN (AEKI)

AEKI have performed Barkhausen measurements on the GRETE fatigued specimens. The testing device was the same used in the testing of irradiated specimens, but the probe have been changed for a probe developed for round specimens. Three tests have been performed on every specimen, and the specimens have been rotated with 120°.

Due to the low ferrite or martensite content of the stainless steel the signal change is low. To reduce the effect of the background noise the signal of the empty probe have been measured, and it have been deducted from the measured curves. The values of the inflexion of the resulted curves were evaluated.

Barkhausen noise may be measured on stainless steel specimens having more than 5 % magnetic phase. In his case correlations seem to exist between the usage factor and the measured results. However the effect of the quantity of the magnetic phase, and the structural effects on the noise value cannot be separated. A simple measurement of the quantity of the magnetic phase or permeability is expected to provide better correlation.

C.3.2.4 Magnetic measurements PSI

Magnetic Susceptibility using a Ferromaster

The magnetic susceptibility χ of all GRETE-specimens was measured using the Ferromaster. Results of measurements on all the fatigue specimens have shown an excellent linear correlation between χ and noc as well as between χ and content of α'. With an adequate knowledge of the material and its load history, it is therefore possible to evaluate the noc and the corresponding usage factor D of a specimen by measuring χ.

Eddy Current Impedance using GMR-Sensors

The determination of martensitic contents by measuring eddy current impedance using very sensitive sensors with a high lateral resolution is a promising technique. After calibrating the system, the direct measurement of the martensitic distribution is possible by scanning the surface. For the investigations the cylindrical part of the specimens was scanned in order to calculate an averaged martensitic content which can be compared with the results from ND.

Remanence field measurements using Fluxgate- and SQUID-Sensors

For the GRETE-fatigue specimens, the remanence field is an appropriate measure for ferromagnetic contents. An advantage of the method is the possibility of performing contactless measurements in relatively large distances.

The expected linear correlation between content of α' and magnetic field strength was proved. In general a linear dependency of the remanence field from the noc was found. The method was found to be very sensitive what is demonstrated for the specimens which formed very small amount of α'.

C.3.2.5 MSM (SPG)

The developed magneto-static measuring procedure represents a simple and appropriate method for non-destructive determination of integral martensite content in austenitic steel and the study of fatigue processes.

The high remanent magnetisation at the ends of some specimen measured is surprising. It prevents a quantitative determination of the martensite concentration in the neck region of the specimen. The reasons for the high content of magnetic phases in the initial state should be investigated. They could be generated by cold work and machining after heat treatment. Also metallurgical reasons might be responsible: The austenite-martensite phase diagram may depend very sensitively from the concentration of some alloying elements and on small contaminations by C or Si. In these cases the martensite concentration may depend very sensitively on the heat treatment technology.

For a quantitative evaluation of fatigue effects the initial state of specimen should be free of martensite. If martensite content in the initial state of specimen cannot be avoided completely, it must be characterised for each specimen individually in order to get precise information on changes as a consequence of fatigue processes.

C.3.2.6 ULRW and USS (VTT)

The material properties are measured with special ultrasonic probe optimised for surface measurement. By combining information from the data measured from large area and calculating areas of interest in the sound path information can be gained to improve potential estimation of degradation levels. The technique is based on combination of three factors: using back scattered ultrasonic signals and induced leaky Rayleigh wave information (1), and simple statistical data analysis (2) in combination with optimised ultrasonic transducer (3). The back-scattered ultrasonic signal is a measure of the amount of reflectors such as micro-pores, inclusions, precipitations, segregations, micro-cracks and cracks as well as of back-scattering from phase boundaries during fatigue damaging and increase of degradation inside the material. The leaky Rayleigh wave component is sensitive to surface properties as known from normal Rayleigh wave probes. Especially cracks cause strong effect on leaky Rayleigh wave. If the crack is deep it cancels the leaky Rayleigh wave signal totally.

The measured data shows quite clearly the correlation to lifetime of measured component and that the increased functional amplitude value is describing the material degradation in LCF-test specimen. The function is chosen in the way, that it is positive growing function with increasing loading cycles. This justifies the physical phenomena in back-scattered ultrasonic signal when material degradation is occurring.

C.3.2.7 HAS (Uni Han.)

For the determination of the state of fatigue, each specimen set has to be measured and calibrated separately, because the generation of martensite depends on the steel grade and is influenced by loading conditions and test temperature. For standard LCF-tests a possibly wide scatter in martensite content is common. Influences on the martensite content can be occur by non-homogeneity of the material and by thermal activated processes on the microstructure.

The state of fatigue could be determined with high accuracy for fatigued specimens at room temperature. A determination of the degradation for fatigued specimens tested at high temperature is difficult. The measuring uncertainty of the martensite content (Neutron Diffraction) is high for one of the set of specimens and the martensite content of two other specimen sets were not available. So it is not possible to purify the trends in harmonic values.

Online measurements during fatigue tests at high temperature seems to be a promising way to determine the fatigue state, because influence quantities, like inhomogeneous microstructure, are eliminated. Another advantage of online measurements is, that the fatigue states respectively usage factors are not limited and usage factor D = 1 could be detected individual for each specimen, because the specimen could be tested until failure.

C.3.2.8 USS (ARCS)

In a first attempt to establish a rating in terms of the degree of LCF induced damage specimens with usage factors of 1.0 and 0.8 has been done. A qualitative damage assessment based on the length, depth and number of cracks observed in C-scan images has been obtained. Whereas strong and moderate indications for cracks are prevailing for specimens with usage factor 1.0 only subtle evidence was found at a value of 0.8; on the other hand there seems to be no correlation between crack formation and the temperature during LCF-testing (20 °C vs 300 °C).

Strong to sufficient evidence of LCF-induced cracks has been found in specimens with usage factor 1.0 by measuring the attenuation of the primary echo during ultrasonic scans with a 20 MHz transducer. Reasonable qualitative agreement between optical inspections and ultrasonic scan images could be obtained; for the usage factor 0.8 only subtle evidence of starting cracks was recognized, none for specimens with lower factors. Due to physical constraints in achieving ideal alignment and "zero" eccentricity over the entire gauge length of all specimens which may be related to (LCF)- induced deformations of the specimen itself or deficiencies in the rotary axis assembly a quantitative assessment of LCF-induced damage is only possible in some selected cases but not for all acquired scan-images; hence a comparison between several specimens scanned and a subsequent ranking could only be established on a qualitative basis.

C.3.3 Conclusion on the measurements

C.3.3.1 Irradiated specimens

Good results were achieved only by NDT techniques that are based on thermoelectric voltage properties or mechanical properties. The TEP (EDF) and STEAM (JRC) measurements have the disadvantage, that the quality of the results is low. Moreover both techniques could not evaluate the recovered state of the annealed Charpy-specimens after irradiation correct, although STEAM has a good correlation with irradiated and irradiated/annealed/irradiated Charpy-specimens. The ABIT (NRI) measurements were carried out only on some groups. But these good achieved results should be transferable to the other groups, because ABIT technique bases on mechanical properties and so should be independent of the steel grade. The ABIT is not completely non-destructive, because a small indentation is left on the specimens.

Inside the group of NDT techniques based on the electromagnetic properties only MBN (Ciemat) achieved good results and MBN (AEKI) achieved partly good results. The main problems for these NDT techniques are different microstructures of the Charpy-specimens, residual stresses, caused by the manufacturing process, or additional residual stresses, caused by the Charpy impact tests. The effect of the embrittlement of the irradiated specimen on the measured values is probably small comparing to the effect of the influence quantities. Regarding these boarder conditions the NDT techniques based on the electromagnetic properties are not reliable for a determination of the damage of RPV steels caused by neutron irradiation at this state.

C.3.3.2 Fatigue specimens

Depending on the chemical composition and its segregations, the mechanical and thermal history, stabilized austenitic stainless steels are prone to phase transformation from the face-centred-cubic austenitic γ-phase to the body-centred-cubic ferromagnetic α'-martensitic phase. The driving force for the phase transformation is energy input due to plastic deformation, cold working and fatigue. The phase transformation is much more pronounced at low than at elevated temperatures and at positions with higher stress concentration, i.e. at locations of larger non-metallic inclusions and/or carbo-nitrides. The more the chemical composition is locally segregated, the more the martensite development and fatigue damage accumulation is strongly localised. This fact evidently makes scanning of the serviced components in critical areas of highly applied mechanical loads to an important NDT task for the early detection of fatigue, i.e. before the initiation of macroscopic cracks takes place. With various NDT techniques, the different states of fatigue have been determined. All the NDT techniques that were used for testing the LCF-specimens have a high spatial resolution and by this the potential to detect strongly localised martensitic phases.

In the GRETE project a quantitatively evaluation of the NDT results has to be performed. In order to get measuring quantities, which are characteristic for the total LCF-specimen either, from the locally measured values were took the mean, or a sensor was used, that allowed an integral testing of the specimen.

In general, good results were achieved by NDT techniques basing on electromagnetic properties as well as ultrasonic properties. The NDT techniques, basing on magnetic properties, and magnetic Barkhausen noise achieved only partly good results. In fact, Barkhausen noise has been revealed to be very sensitive to internal stresses in the martensite phase, which is a disadvantage for measuring martensite content. But in turn this sensitivity could be valuable for characterising such internal stresses. Although there is no NDT technique, getting good results for all different groups of fatigue, there is at least one NDT-technique, which is able to determine the state of fatigue of each group very well.

Inside the group of NDT techniques based on the electromagnetic properties the best results were achieved by IZFP (GMR) and Uni-Hannover (HAS). The Uni-Hannover got no clear trend in measured values for LCF-specimens tested at test temperature 300°C. For group b the results of IZFP are only satisfactory. A promising solution to understand the effects of fatigue on the material at elevated temperatures is online measurements during the fatigue tests. Both techniques are able to perform these online measurements.

The other NDT techniques based on the electromagnetic properties as well as techniques based on magnetic properties achieved only partly good results because of various reasons that are described in detail in the final report. In most cases the low martensite contents respectively the low content of generated martensite is the main reason for problems in determination of the fatigue state of the LCF-specimens. The comparison of both GMR measurements, carried out by IZFP and PSI, show differences in the results that have to be investigated.

Inside the group of NDT techniques based on ultrasonic testing VTT (backscattering, ULRW) achieved very good results. Only for group h a correlation between measured values and state of fatigue could not be found. In comparison to backscattering technique the pulse-echo technique of ARCS is only suitable for crack detection.

C.4 Comparison of the performance of the non destructive techniques (WP7)

C.4.1 Ranking process

A specific methodology has been developed in order to compare the different methods. The ranking process evaluated three main parts: the results of the analysis, the quality of the results and the on-side applicability. With the scored analysis results and scored quality of the analysis results an evaluation of the NDT results was possible. The on-site applicability is divided into three additional parts that help to draw recommendations inside WP8 for the industrial use of the different NDT techniques.

All the results were analyse by divided each kind of criteria in six categories from 0 (corresponding for instance to 0 correlation or impossible for on-site suitability) to 5 (corresponding for instance to 100 % for coefficient or very easy for handling of the device).

C.4.1.1 Analysis results

The results of the analysis should reflect the functional relationship between measured values and required quantities. For the analysis of the achieved results for fatigued LCF-specimens and irradiated Charpy specimens, the specimen sets were separated in different groups according to their composition, fatigue testing condition or irradiation level. The required quantities for LCF-specimen were the number of cycles as a minimum and additional the martensite content and for Charpy-specimen the transition temperature as a minimum and additional the fluence.

For simple analysis methods the achieved results respectively the correlation coefficient for each group were scored in %. For complex analysis methods the calculated standard deviation of the test set is referred to the range of measured values of the test set. The achieved results for each group of specimens were scored in % of the referred standard deviation.

C.4.1.2 Quality of the NDT results

The repeatability standard deviation referred to the range of measured values for a group of fatigued respectively irradiated specimens was an estimation for the measuring uncertainty. Therefore the standard deviation was calculated from at least 10 repeat measurements. The standard deviation of the repeat measurements should be referred to the range of measured values of the group; the specimen for repeat measurements was taken off.

C.4.1.3 On-site applicability

The on-side applicability was divided into the following three points:

- Handling of device and technique during carried out measurements including the complexity of device handling and installation methods, the special requirements for component and local environment (access, surface finish, temperature, humidity, etc).
- On-site suitability including the durability/service life of device in site environment, the possible modes of operation (on line measurement, periodic measurement during outages, tests on components in situ).
- Costs of NDT-technique, including the future development costs and timescales, the maintenance costs and the costs of device.

C.4.1.4 Summary of ranking results

Finally, all the ranking results were joined in a bar graph. By presenting the results in this way, the strengths and the weaknesses of the technique can be estimated at the first sight. Furthermore an easy comparison between the different NDT techniques is possible.

The presentation of the achieved results in such a compressed and compact way enables a client to get an overview of the various NDT techniques and their potential for industrial application.

Example of representation of the results:

5												
4												
3												
2												
1												
Group	**a**	**b**	**c**	**d**	**e**	**f**	**g**	**h**				
Score	Analysis results								Quality of results	Handling	Adapt-ability	Costs

C.4.2 Comparison of the non-destructive techniques on the irradiated specimens

Analysis of Results

Good results were only obtained with techniques based on measurement of ThermoElectric Power (TEP and STEAM) or mechanical properties (ABIT). Of the electromagnetic techniques tested, only MBN achieved partly good results. For the other electromagnetic techniques, it was concluded that the effects of neutron irradiation embrittlement in these steels was partly obscured by other influencing parameters such as the plastic deformation and residual stresses in the faces of the broken Charpy specimens used in the round-robin exercise.

Quality of Results

For the thermoelectric power measurements, repeatability of results was low. Only MBN showed good quality of results.

On-site Applicability

Only TEP/STEAM and MBN techniques had reasonable ranking in this area combined with good Quality of Results. Unfortunately those techniques which ranked highest regarding On-site Applicability (3MA, HAS) demonstrated the poorest capability regarding the Quality of Results.

For the fatigue monitoring:

- None of the NDT techniques was able to find any meaningful correlation between the measured NDT parameters and the irradiation damage in specimens which had been irradiated, annealed and reirradiated. This illustrated the complex nature of the microstructural and material property changes occurring in these specimens.

- Differences in irradiation history between groups of specimens appear to have significant effects on the NDT measurements and the resulting correlations.

C.4.3 Comparison of the non-destructive techniques on the fatigue specimens

Analysis of Results

Regarding analysis of results, electromagnetic (GMR, HAS, Ferromaster) on different groups of fatigue specimens i.e. good correlations were achieved between the measured electromagnetic or ultrasonic parameter and the evolution of the microstructure as measured by the total number of fatigue cycles seen by each specimen. Ultrasonic (ULRW and USS) techniques achieved good results on the detection of fatigue damage. Techniques based on magnetic properties (Fluxgate), Magnetic Barkhausen Noise and Multi Parameter Techniques (3MA) achieved only partly good results. Although there is no NDT technique that achieves good results for all different specimen groups, there is always one technique that is able to determine the state of fatigue in each group of specimens. In particular:

- In the steels studied in the GRETE project, all the electromagnetic techniques were sensitive to the amount of ferromagnetic martensite phase generated in each material as a result of low cycle fatigue. The varying performance of each technique between different steels and the same steels fatigued at different temperatures was linked directly to the different amounts of martensite present. It should be noted that martensite content is only an indirect measurement of the fatigue damage accumulation influencing the materials mechanical properties relevant to component structural integrity issues.
- The Magnetic Barkhausen Noise measurement technique, in addition to being sensitive to martensite content was also shown sensitive to residual stresses in the steel. Thus it was recognised that its use would be most effective for fatigue monitoring as part of a combination of complementary techniques, rather than on its own.
- Ultrasonic LRW and backscatter techniques exhibited a non-linear correlation with accumulated fatigue damage; however, only a weak dependence was observed up to 0.6 usage factor, followed by a stronger dependence at higher usage factors.
- It was noted that nominally similar electromagnetic and ultrasonic techniques used by different laboratories sometimes produced significantly different results. This was taken as evidence of the need for optimised experimental and system design in order to achieve consistent results.
- All techniques required calibration on a set of fatigue specimens spanning the full fatigue usage factor, if quantitative information on accumulated fatigue damage was to be obtained in these blind round-robin tests. The amount and effectiveness of the ‘training’ required of the technique in this way, and the complexity of the analysis process needed to extract the fatigue accumulation on blind specimens, differed between techniques.
- As the number of specimens within any Group was relatively low, it was not possible to get statistically valid data on which to calibrate the techniques.

Quality of Results

All techniques, except ultrasonic LRW technique, which performed well on Analysis of Results also demonstrated high Quality of Results, indicating good repeatability with the systems employed.

On-site Applicability

Whilst, this is the most subjective of performance indicators, the ranking achieved indicated that all but the permanently laboratory based techniques (Fluxgate) offered the potential for on site use. However specific factors (such susceptibility to vibration, temperature, humidity, shock, etc) affecting equipment durability in hostile conditions were not addressed. If they had been, it is likely that the scores for most existing techniques and systems would have been lower than reported.

D. RECOMMENDATIONS (WP 8)

The round-robin trials performed on fatigued stainless steel specimens and irradiated ferritic steel specimens have successfully identified NDT techniques with good potential for monitoring degradation processes in certain materials. However, assessment of the industrial feasibility of these NDT techniques requires further systematic studies of the capability and reliability of these techniques under conditions which more accurately reflect the industrial environment. A series of tests are necessary to take the most promising techniques to the point where they can be used on industrial plant components. These tests are developed below for fatigue damage and irradiation embrittlement monitoring.

D.1.1 Continuous or periodic monitoring studies on single specimens/components

It is probable that some of the scatter in the data observed in the GRETE studies arose from specimen to specimen variations. If these techniques were used on industrial plant only one specimen (the component being monitored) would be used and it would be monitored continuously or intermittently throughout its operating lifetime. Monitoring the damage accumulation in single specimens as the damage is accumulated will require tests to be performed: for the fatigue specimens periodically or continuously as the specimen is fatigued in the laboratory and for irradiation specimens periodically between periods of specimen irradiation with a Materials Testing Reactor.

D.1.2 Studies on materials experiencing realistic plant operational transients and thermo-mechanical fatigue

During the GRETE programme tests have all been performed on specimens that have been mechanical fatigued, rather than those that have experienced fatigue. On nuclear power plant, components experience thermo-mechanical fatigue. It is important therefore to test the ability of promising NDT fatigue monitoring techniques on specimens undergoing thermo-mechanical fatigue. Initial laboratory studies aimed at resolving the effects of temperature and stress cycling would be valuable.

D.1.3 Studies on materials that do not generate ferromagnetic martensite during fatiguing

For fatigue monitoring, a further important step would be to test the capabilities of the most promising NDT techniques to monitor directly the changes in materials properties responsible for changes in materials mechanical properties. This would require tests to be performed on other relevant stainless steels which do not generate a significant ferromagnetic martensite phase during fatiguing, since for electromagnetic techniques at least this change dominates the other possible effects.

D.1.4 Studies on industrial size components

Finally, for both fatigue and irradiation embrittlement monitoring systems practical tests need to be performed on truly representative thickness and shapes of materials to confirm that techniques which have shown good capabilities in ideal laboratory conditions on small specimens can be developed for use on realistic components.

E. CONCLUSION

In the GRETE programme, electromagnetic (GMR, HAS, Ferromaster and possibly 3MA) and ultrasonic (LRW and backscatter) techniques showed the most promise as methods for monitoring fatigue damage accumulation in austenitic steel piping. The TEP/STEAM and ABIT techniques showed the most promise as methods for monitoring neutron irradiation embrittlement in reactor pressure vessel steels. More generally:

- The GRETE round-robin tests on well characterised stainless steel specimens fatigued to different fatigue usage factors provided an effective test of the basic capabilities of the different NDT techniques on the range of stainless steels (AISI 304L, AISI 347 and AISI 321) fatigued at nominally room temperature and 300ºC.
- The round-robin tests on fatigue specimens were only relevant to steels that generate a ferromagnetic martensite phase during fatiguing. As the amount of ferromagnetic martensite generated was a strong function of specimen temperature, techniques performed better at room temperature than 300ºC.
- The round-robin tests did not demonstrate the capabilities of the NDT techniques to monitor the accumulation of fatigue damage directly in a single specimen or specific location on a plant component, the latter being the more realistic situation and requirement on an industrial plant.
- The round-robin tests on broken Charpy impact specimens from specimens irradiated in different European reactors provided a less effective test of the capabilities of the different NDT techniques for monitoring irradiation embrittlement in RPV steels. This was due in part to the complex irradiation history of some of the specimens and the additional damage introduced into the specimens during the earlier Charpy tests.
- The round-robin tests did not provide a test of the capabilities of the NDT techniques to monitor the accumulation of irradiation damage in a single specimen or specific location on a plant component, which is the more realistic situation and requirement on a nuclear plant.

The aim of the GRETE project was to assess the capability and the reliability of some innovative non-destructive techniques. These techniques were tested for an evaluation of neutron irradiation damage in reactor pressure vessel steel and an evaluation of fatigue damage of piping. Series of aged samples have been characterised metallurgically and mechanically before being tested by the participants using various non-destructive techniques during round robin exercises.

To summarise, the following objectives have been achieved through the course of this project:

1. Observation of the microstructural changes due to damage in order to understand the NDT signals for the fatigue specimens.

2. Evaluation of the sensitivity of the techniques (round-robin exercise) to quantify the changes in the signal due to aging (neutron irradiation, fatigue).

3. Establishment of a relationship between the signal and the mechanical characteristics in order to give engineers quantitative data for evaluation of the remaining lifetime: These

relationships have been determined for the techniques which show good correlation between the non destructive signal and the damage parameter (% of martensite, noc, fluence, transition temperature) though correlation curves.

4. Evaluation of possible technology transfer towards the industry for practical use of these techniques: recommendations have been established.

The GRETE project has enabled to compare promising innovative non-destructive techniques on well-characterised reference specimens for the monitoring of irradiation damage in RPV steel and fatigue of austenitic piping. The collaboration of European teams in the field of non destructive evaluation of component aging has paved the way for development and qualification of such techniques in the nuclear power plants.

TABLES

Technique	Acronym	Partner
Thermopower measurements	TEP	EDF-JRC
Micromagnetic measurement	MAG	IZFP
Magnetic Barkhausen noise	MBN	IZFP-CIEMAT-AEKI
Harmonic Analysis of magnetic signals System	HAS	UNI – HAN.
Automated Ball Indenter	ABI	UJV REZ

Table I: Non-destructive techniques performed on irradiated samples

Technique	Acronym	Partner
Eddy current impedance with Giant Magnetic Resistance sensor	GMR	IZFP, PSI
Magnetic Barkhausen noise	MBN	EDF-AEKI
Harmonic Analysis of magnetic signals System	HAS	UNI – HAN.
Permeability measuring with Ferromaster	Ferromaster	PSI
Magneto static measurement	MSM	SPG
Remanence field measuring with fluxgate	3-axis fluxgate	PSI
Ultrasonic Leaky Raleigh Wave technique	ULRW	VTT
Ultrasonic Scattering or Backscattering	USS	VTT-ARCS

Table II: Non-destructive techniques performed on low-cycle fatigue specimens

FIGURES

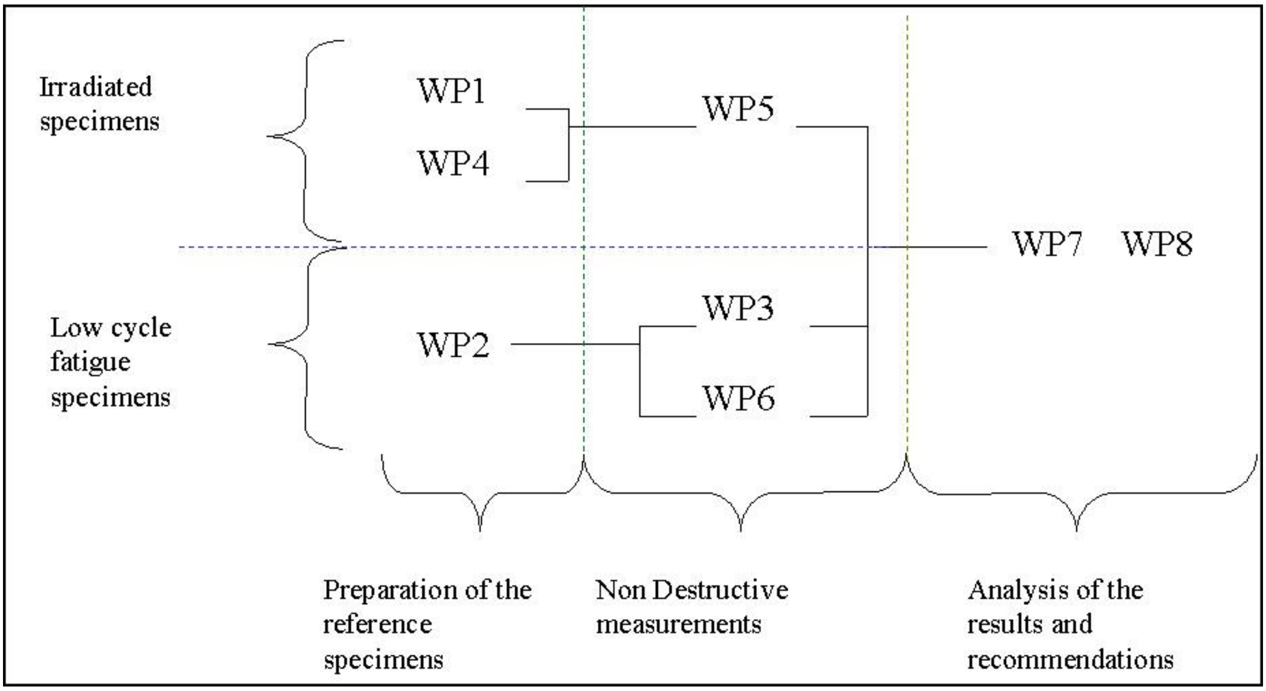

Figure 1: Synthetic organisation of the work packages

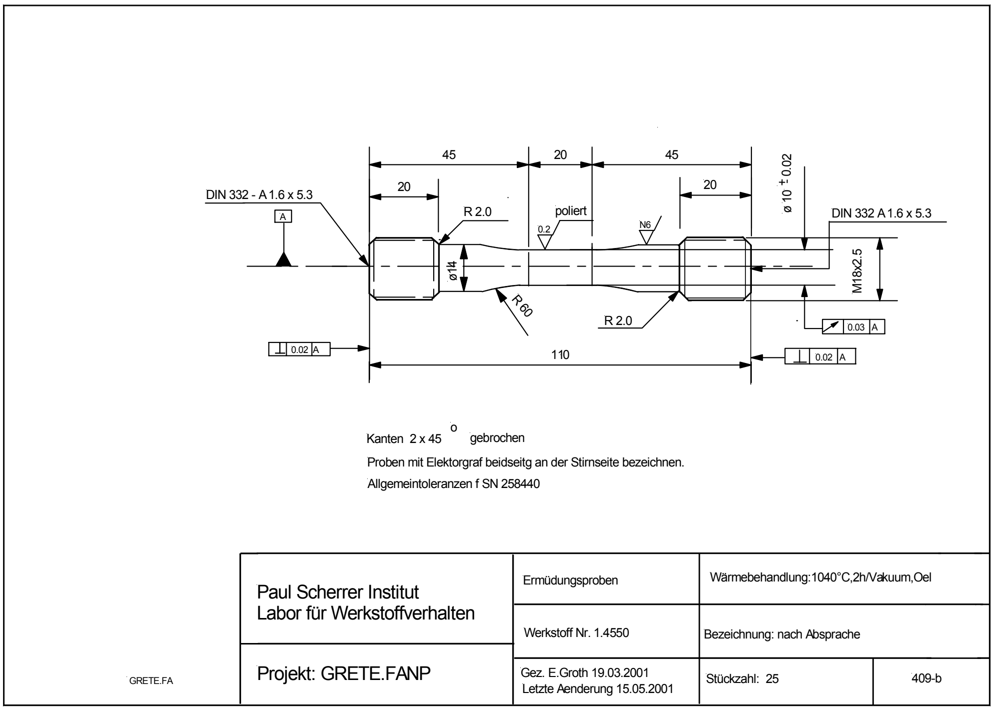

Figure 2: Sample for LCF-tests